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CONTINUED STUDY OF ADVANCED-TEMPERATURE NICKEL-BASE ALLOYS TO INVESTIGATE VANADIUM ADDITIVES

By John C. Freche, Thomas J. Riley and William J. Waters

Lewis Research Center
Cleveland, Ohio

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SUMMARY

An investigation was conducted to extend the useful temperature range of a series of previously developed advanced-temperature nickel-base alloys having a basic composition of 79 percent nickel, 8 percent molybdenum, 6 percent chromium, 6 percent aluminum, and 1 percent zirconium.

As-cast modifications of the basic composition utilizing vanadium and carbon, as well as tungsten, vanadium, and carbon, provided improved rupture and impact properties.

The strongest composition, a tungsten-vanadium-carbon modification of the basic alloy, displayed 768-, 301-, and 101-hour as-cast rupture lives at 15,000-psi stress and temperatures of 1800°, 1850°, and 1900° F, respectively, in single tests.

All of the alloys demonstrated good impact resistance. Most of them exceeded 62.5 inch-pounds impact resistance.

On the basis of swaging and rolling tests, it must be concluded that the alloys developed to date are essentially usable only as cast alloys, although a limited degree of swageability was demonstrated in a few instances. There was no evidence of catastrophic oxidation in the vanadium-bearing alloys after prolonged stress-rupture testing at 1800°, 1850°, and 1900° F in air. None of the alloys required vacuum-melting techniques. All were melted under an inert gas blanket and were poured in air.

INTRODUCTION

The demand for materials having higher load-carrying capacity at ever higher temperatures continues unabated in all phases of modern technology. In many instances, significant technical advances can be made by only slight improvements in material properties. One example of this is turbine engines, where substantial increases in jet-engine thrust can be achieved by permitting increases in turbine-bucket operating temperatures of only 100° F.

The problem of obtaining increased stress-rupture life at elevated temperatures is frequently complicated by additional requirements and these usually depend on the intended application. For example, in turbine-engine powerplant applications such as turbine buckets, good oxidation and impact resistance are other major requirements that must be met simultaneously in order to provide a satisfactory material. Thus, the intended application frequently indicates the possible metal systems that may be profitably investigated. One of the more promising methods of achieving the material properties needed for the applications just cited lies in extending the useful temperature range of the so-called nickel or cobalt-base superalloys. Consequently, an investigation to provide an advanced-temperature nickel-base alloy was initiated at the Lewis Research Center.

E-532

Reference 1 describes the results of the initial phase of this investigation in which a series of nickel-base alloys with good stress-rupture, oxidation-resistance, and impact properties was developed. The basic composition, which is (by weight) 79 nickel, 8 molybdenum, 6 chromium, 6 aluminum, and 1 zirconium, was modified by small additions of boron, carbon, titanium, and titanium plus carbon. Nickel content was adjusted to account for these additions. The properties of the basic alloy were generally enhanced by these additions. It was believed that such mechanisms as complex-carbide hardening, dispersion hardening through intermetallic compound formation, and possibly solution hardening were acting. The strongest composition (a titanium-plus-carbon modification of the basic alloy) displayed rupture lives in the as-cast condition of 380 hours at 1800° F and 107 hours at 1850° F with a 15,000-psi stress. These data compared favorably with those obtained with the latest commercially available superalloys (ref. 2).

The present report describes a continuation of the investigation of the basic alloy system developed in reference 1. Attempts were made to achieve greater strength at elevated temperatures primarily by utilizing vanadium as an alloying constituent. It was known that vanadium is a strong carbide former, and it was believed that vanadium might also act as an alloy strengthener in a manner analogous to titanium. Another consideration in the present investigation was that of achieving further improvement in alloy properties by the use of tungsten as a possible matrix strengthener in addition to molybdenum.

The alloys were evaluated on the same bases as in reference 1, namely, stress-to-rupture life, impact resistance, and formability. Attempts were made to broaden the scope of the formability evaluation by including rolling trials as well as the swaging attempts. Short-time tensile test data at room temperature and at 1800° F were also obtained for the most promising compositions. Hardness data were obtained for most of the compositions investigated. Melts were made by high-frequency induction heating under an argon blanket. Investment-casting techniques were employed in order to eliminate the need for extensive machining of test samples.

MATERIALS, APPARATUS, AND PROCEDURE

Alloys Investigated

The basic alloy as described in reference 1 had a nominal composition by weight of 79 percent nickel, 8 percent molybdenum, 6 percent chromium, 6 percent aluminum, and 1 percent zirconium. As is evident from table I, which lists the nominal compositions of the alloys considered in this investigation, the basic alloy was modified by vanadium, carbon, and tungsten additions. The vanadium and carbon additions were made by reducing the weight percentage of nickel by an equal amount. As an example of the nomenclature used, a 2.5-percent-vanadium and 0.125-percent-carbon addition to the basic alloy is referred to in the table and in the text as "basic + 2.5 V + 0.125 C." The tungsten modifications of the basic alloy were made by reducing the weight percentage of molybdenum an equal amount. An example of the nomenclature used to describe the tungsten modified alloys is the designation "Mo-2-W." This means that the basic alloy was modified by adding 2 weight percent tungsten and removing 2 weight percent molybdenum.

Randomly selected heats of most of the compositions investigated were chemically analyzed by an independent laboratory. The analyses are shown in table II. Comparison of tables I and II indicates that the major degree of variation between melts and from the nominal composition occurred with the element zirconium. As in the investigation of reference 1, zirconium was not added as a melting constituent but rather was picked up from the crucible during the melting process. Although the same care was exercised in maintaining quality control of castings as in the investigation of reference 1, a greater variation in zirconium content occurred. This may have been due to unavoidable variations in melting times that did occur occasionally, although the average exposure time was 20 minutes. It should be noted that the 20-minute melting time was a function of a variety of factors including melt size, melting constituents, crucible material, and induction-unit characteristics.

The percent purities of the various alloying elements used, as determined by the suppliers, were as follows:

Nickel	99.95+
Electrolytic chromium	99.5+
Molybdenum	99.0+
1100 Aluminum	99.0+
Vanadium	99.8+
Tungsten	99.9+

Casting Techniques

The casting techniques employed were similar to those described in reference 1. They are briefly discussed in the following sections.

Wax patterns. - Expendable wax patterns of the stress-rupture and swage bars were prepared to final test dimensions in precision dies and were assembled as shown in figure 1. A wax pattern (2 by 1/2 by 1/4 in.) was substituted for the swage-bar pattern in several instances in order to provide rolling samples. Only the impact-bar pattern was made slightly oversize. This was done to permit finish machining to exact tolerances, so that the test samples could all be gripped identically in the flat jaws of the Izod impact tester.

Mold preparation. - The wax pattern was invested in a silica slurry with a commercial binder. The investment was allowed to settle, and was dried and placed in a furnace to melt out the wax patterns. Furnace temperature was 200° F upon insertion of the investment molds, and a program of gradually increasing temperature was initiated. The maximum furnace temperature attained was 1800° F. Several hours before casting, the mold temperature was dropped to 1600° F, and all melts were poured into 1600° F molds.

Melting procedure. - A 50-kilowatt, 10,000-cps water-cooled induction unit was used for melting. Melts were made in stabilized zirconia crucibles under an inert gas (commercially pure argon) blanket. In casting, the crucible was first charged with nickel platelets. Vanadium, carbon, and tungsten additives were in the form of powders that previously had been compressed inside aluminum-foil containers. These were inserted into the crucible in such a manner that the nickel platelets completely surrounded and covered them, and thereby minimized the quantity of these powders lost during melting. After this charge had melted, chromium platelets were introduced; and, when these had been absorbed in the melt, molybdenum chips were added. About 3 minutes were allowed to permit the molybdenum to be dissolved satisfactorily. The necessary quantity of aluminum required to make up the final composition was added last in the form of slugs. The melt was poured within 30 seconds after the final aluminum additions in order to minimize the loss of aluminum by vaporization. As indicated previously, zirconium was absorbed from the crucible during melting. Stabilized zirconia crucibles were used; it was intended by this means to take advantage of the beneficial effect of minor zirconium additions to nickel-base alloys that frequently has been observed and is reported in references 3 and 4. The procedures described to make the alloying additions were evolved as a result of several attempts and were found to be the most satisfactory. The total weight of material per melt was approximately 760 grams.

Pouring temperature, determined by optical pyrometer measurements, was 3150° ±50° F for all melts. All melts were top-poured into 1600° F molds (which gave a satisfactory grain size) without the inert-gas-blanket coverage maintained during melting, and were permitted to cool slowly to room temperature (overnight) before removing the investment.

Inspection Procedures

All cast samples were vapor-blasted to a smooth surface before being inspected. The stress-rupture and swage bars were subjected to both radiographic and zyglo inspection before testing. The impact bars were first radiographed, then machined, and zyglo-inspected after machining. Samples with significant flaws such as cracks or extensive porosity, as revealed by the inspection methods, were eliminated from further processing or testing.

Heat Treatment

All the alloys except some of those with tungsten additions were given a homogenization treatment so that comparison tests with the as-cast samples might be run. "Homogenization" in the complete sense was not achieved at the relatively low temperatures employed. However, partial homogenization of the structures was achieved, and this term therefore is used throughout this report. The homogenizing time and temperature (16 hr at 2000° F) and method (furnace-heated under argon atmosphere and air-cooled) were the same as those given the titanium and titanium-plus-carbon modifications of the basic alloy investigated in reference 1. Microexamination of small samples of each composition so treated revealed no evidence of grain-boundary melting. However, no study was made to determine the optimum homogenization treatment for these alloys. In the light of the test results and because heat treatment sometimes enhances the properties of this type alloy (ref. 1), it would appear that further studies of this type might be warranted.

Alloy Physical Property Determinations

Stress rupture and tensile tests. - Stress-rupture tests were run with all of the vanadium-carbon modifications of the basic alloy at

15,000 psi and 1800° F. These stress and temperature levels were the same as those used in reference 1, and equaled or exceeded the temperature and stress requirements of turbine buckets in most current turbojet engines. Additional stress-rupture tests at 1850° and 1900° F were run with the strongest vanadium and carbon modified alloy. The tungsten modified alloys were, for the most part, evaluated at 1900° F. In all cases the stress levels were set at 15,000 psi. Most of the alloys investigated were run in both the as-cast and homogenized conditions. The exact conditions for all the stress-rupture tests are listed in table III.

Tensile tests were run at both room temperature and 1800° F with the most promising modifications of the basic alloy in the as-cast condition. At least two tests were run at each temperature for each alloy. The tensile test conditions for all the alloys investigated are summarized in table IV. Figure 2 shows the type of test bar used for both stress-rupture and tensile tests.

Impact tests. - Laboratory impact tests were performed for most of the alloys in both the as-cast and homogenized conditions with a low-capacity Bell Telephone Laboratory Izod Impact Tester; this unit is described in reference 5. Tests were run with two specimens in both the as-cast and homogenized conditions. Two of the better impact-resistant commercial alloys (commercially cast) were also tested in the as-cast condition for purposes of comparison. A summary of the test conditions and the alloys investigated is given in table V. Impact bars were 3/16 by 3/16 by $1\frac{1}{2}$ inches and were unnotched. Test bars were inserted between the grips of the tester to a depth of 1/2 inch, and the point of impact of the pendulum was 1/8 inch from the free end of the bar. Total capacity of the pendulum was 62.5 inch-pounds, and the striking velocity was approximately 135 inches per second.

Workability. - Workability of the alloys was determined primarily by swaging. Attempts were made to swage most of the compositions (as-cast) at elevated temperatures. The alloys tested and the swaging conditions are summarized in table VI. In swaging, bars approximately 0.520 inch in diameter by $1\frac{1}{2}$ inches long were fed through a 0.500-inch die. This provided a reduction in area of 7.5 percent. The specimens were heated in an induction furnace adjacent to the swager. Specimen temperature was measured by an optical pyrometer while the specimen was still in the furnace. When the test temperature was attained, the specimen was removed from the furnace with tongs and inserted into the swager for approximately 5 seconds. As in reference 1, visual inspection for cracks was usually sufficient to determine lack of workability. When no cracks were apparent, the bars were zyglo-inspected.

In order to further evaluate workability of one of the strongest alloys, an attempt was made to hot-roll small as-cast slabs (1 by 1/2 by

1/4 in.) at various temperatures. Table VII lists the rolling conditions used. The specimens were heated in an induction-tube furnace immediately adjacent to the rolls. Specimen temperature was measured with a thermocouple that was in direct contact with the specimen. When the desired temperature was reached, the specimen was ejected from the furnace onto a small chute that conveyed it directly to the rolls. The chute was employed because the small specimen tended to lose heat rapidly and had to be transferred quickly from the furnace to the rolls. Both specimen temperature and percent reduction were varied in an attempt to find satisfactory rolling conditions.

Metallographic studies. - Metallographic samples were made for each alloy in both the as-cast and homogenized conditions. Photomicrographs of the two strongest alloys are presented herein.

Hardness determinations. - Hardness data were obtained with a Rockwell hardness tester for most of the alloys, in both the as-cast and homogenized conditions. Table VIII lists the alloys so evaluated. Samples for hardness tests were obtained by cutting 1/4-inch slabs from unused impact bars.

RESULTS AND DISCUSSION

Stress-Rupture Data

A major method of evaluating the alloys evolved in this investigation was by stress-rupture tests. All of the stress-rupture results are listed in table III.

Basic alloy and basic alloy plus carbon. - The 1800° F, 15,000-psi data for the basic NASA alloy as well as the 0.125- and 0.250-percent-carbon modifications of this alloy form the base lines for comparison with the various alloys investigated herein. These data were obtained in the investigation of reference 1, but for convenience they are also listed in table III. The basic alloy data show approximately 19- and 46-hour life for the as-cast and homogenized conditions, respectively. The 0.125-percent-carbon modification of the basic alloy demonstrated 73- and 133-hour life, while the 0.250-percent-carbon modification showed 113- and 131-hour life in the as-cast and homogenized conditions.

Basic alloy plus vanadium and carbon additions. - The effects of combined vanadium and carbon additions to the basic alloy on 1800° F, 15,000-psi life are shown in figures 3 and 4. Figure 3 shows the effect on rupture life caused by adding various nominal percentages of vanadium (up to 5 percent) while maintaining a constant carbon content of 0.125 percent. A maximum life of 560 hours was attained in the as-cast condition for a 2.5-percent-vanadium addition. The homogenization treatment did not improve test life, which was as much as 180 hours below the as-cast life for some of the compositions. Figure 4 shows the effect on 1800° F, 15,000-psi rupture life of adding varying percentages of vanadium while maintaining a constant carbon content of 0.250 percent. The peak life values in this case were obtained at the highest percentages of vanadium considered. The maximum as-cast life was 430 hours and occurred at a 5-percent-vanadium addition. Again, the homogenization treatment was, generally speaking, not beneficial. Below a 1.5-percent-vanadium addition, a slight improvement in rupture life was obtained.

Comparison with the data obtained in reference 1 indicates that a substantial improvement in as-cast rupture life was achieved in the present investigation by utilizing combined vanadium and carbon additions to the basic alloy. The maximum as-cast 1800° F, 15,000-psi rupture life in reference 1 was 380 hours and occurred with a 1.5-percent titanium plus 0.125-percent-carbon modification of the basic alloy (basic + 1.5 Ti + 0.125 C). The 565-hour life attained with the 2.5-percent-vanadium plus 0.125-percent-carbon modification (basic + 2.5 V + 0.125 C) greatly exceeds this value. It is apparent from the foregoing that excellent as-cast rupture properties (better than those obtainable with titanium and carbon) can be achieved by combined vanadium and carbon additions to the basic alloy. It is also possible that further improvements in rupture life may be obtained by use of a different heat treatment. Further studies to determine the optimum heat treatment for a specified test condition would appear to be warranted, particularly since the 1800° F, 15,000-psi rupture life of the titanium-plus-carbon modification of the basic alloy (ref. 1) was so improved (380 to 574 hr) after heat treatment.

Alloys with tungsten modifications. - Another investigative approach used was to add tungsten in varying amounts at the expense of molybdenum, always maintaining a total tungsten-plus-molybdenum content of 8 weight percent. One such attempt was made with the basic alloy composition by providing equal weight percentages of tungsten and molybdenum (alloy Mo-4-W). No other compositional changes were simultaneously attempted. No significant improvement over the basic alloy in the 1800° F, 15,000-psi rupture properties was obtained by this change (see table III). Actually, the as-cast life was improved slightly from 19 to 26 hours, but the life of the homogenized sample decreased from 46 to 14 hours.

The procedure of adding tungsten in various percentages while maintaining a total tungsten-plus-molybdenum content of 8 weight percent was also applied to the strongest vanadium-plus-carbon modification of the basic alloy (basic + 2.5 V + 0.125 C). The strengthening effect of this procedure was first evaluated by stress-rupture tests at 1900° F and 15,000-psi stress. These tests were conducted at this higher temperature not only because the goal of the investigation was to extend the temperature capability of these alloys but also because of the much shorter testing times involved. Table III indicates that the maximum life at 1900° F was 101 hours and was obtained with alloy Mo-4-W + 2.5 V + 0.125 C. This alloy was subsequently tested at 1800° F with a 15,000-psi stress. At 1800° F an as-cast life of 768 hours was attained, and at 1850° F a 301-hour life was demonstrated (table III). This 1800° F life represents approximately a 25-percent improvement over the strongest vanadium-carbon alloy (basic + 2.5 V + 0.125 C) discussed previously.

It is apparent from the foregoing that, although the addition of tungsten to the basic alloy had no noticeable effect on rupture life, an appreciable beneficial effect was achieved when a similar addition was made to the strongest vanadium-plus-carbon modification of the basic alloy. If the additional solution straining due to the partial substitution of tungsten for molybdenum were a major strengthening factor, both the basic alloy and the vanadium-plus-carbon modified alloy might be expected to show improved life characteristics. The beneficial effect derived in the case of the vanadium-plus-carbon alloy therefore is concluded to be primarily due to mechanisms other than solution straining.

Stress-rupture comparisons with current high-temperature alloys. - The previous curves indicate that rather high stress-rupture lives are obtainable with some of the modifications of the basic NASA alloy considered in this investigation. Figure 5 provides an as-cast stress-rupture life comparison at 15,000 psi of the strongest NASA alloy (Mo-4-W + 2.5 V + 0.125 C) evolved in this investigation, the strongest modification of the basic NASA alloy (basic + 1.5 Ti + 0.125 C) described in reference 1, and two of the strongest commercial high-temperature nickel-base alloys. The commercial alloy data were obtained from data folders of the Westinghouse Corporation and the Kelsey-Hayes Co. The tungsten-vanadium-carbon modification of the basic NASA alloy shows a slight improvement over the stronger of the two commercial alloys for the temperature range considered, from 1800° to 1900° F. Because of the extensive operating time required at temperatures below 1800° F and 15,000-psi stress, no attempts were made to extend the tests below this temperature.

It should be noted that this comparison is based on single test results for the NASA alloy as against a probable large amount of data for the commercial alloys. It is made only to provide an indication of the potential of the NASA alloys. If these alloys are to be considered for production, many more tests must be made to provide a complete evaluation, and a casting procedure suitable for production practice evolved.

Tensile Test Data

Table IV summarizes both room temperature and 1800° F as-cast tensile test data for the vanadium-carbon modified basic alloy with the highest stress-rupture life (basic + 2.5 V + 0.125 C) and the tungsten-vanadium-carbon modified basic alloy with the highest stress-rupture life (alloy Mo-4-W + 2.5 V + 0.125 C). Ultimate strength, percent elongation, and percent reduction in area are listed. The values reported are averages of at least two runs at each condition. Also included in the table for convenient comparison are tensile test data for two of the highest rupture strength, high-temperature nickel-base alloys, Nicrotung and wrought Udimet 700. These latter data were obtained from data folders of the Westinghouse Corporation and the Kelsey-Hayes Co., respectively. In comparing ultimate-strength values, the tungsten-vanadium-carbon modified basic NASA alloy shows approximately a 10-percent improvement over the vanadium-carbon basic alloy modification. At the same time, however, the former alloy also shows lower percent elongations and reductions in area.

Comparison of the NASA alloys with the commercial alloys listed indicates a remarkable similarity between the tungsten-vanadium-carbon modified NASA basic alloy and Nicrotung in all the tensile properties considered. Forged Udimet 700 displayed much higher percent elongations than either of the NASA alloys. This was to be expected because it is a wrought product. The Udimet alloy also demonstrated almost 100 percent greater room-temperature strength than either of the NASA alloys. Its 1800° F strength, however, was lower.

E-532

Impact Resistance

The impact resistance data for the NASA alloys considered herein, as well as that obtained with two other high-temperature alloys, are listed in table V. All the data obtained are reported. Average values are not given because in many cases the samples could not be broken in the low-capacity Bell Telephone Izod tester. When the samples did not break, the impact resistance values were merely listed as exceeding 62.5 inch-pounds. Most of the NASA compositions shown have good impact resistance. A trend of decreasing impact resistance with increasing vanadium content for the higher carbon content considered (0.250 percent) is apparent. Samples of the strongest modification of the basic alloy (Mo-4-W + 2.5 V + 0.125 C) could not be broken. The 16-hour, 2000° F homogenization treatment did not significantly alter the impact resistance. The two commercial alloys tested also showed good impact resistance. The alloy X-40 demonstrated slightly less than 62.5 inch-pounds impact resistance, and Nicrotung was comparable with the tungsten-vanadium-carbon modification of the basic NASA alloy in that it also could not be broken in this test. It should be noted that Nicrotung was tested, since it represented one of the strongest commercial nickel-base alloys. The X-40 alloy (although not nickel-base) was tested because it had previously demonstrated very good impact resistance in notched bar tests (ref. 5). By testing X-40 in exactly the same manner as the alloys developed in this investigation, it was possible to make direct comparisons.

All the compositions utilizing vanadium and carbon show substantial improvement in impact resistance over the strongest alloy (basic + 1.5 Ti + 0.125 C) reported in reference 1, which had approximately 40 inch-pounds impact resistance. Thus, it appears that the vanadium additions are beneficial from the standpoint of providing increased impact resistance as well as increased rupture life.

Workability

The results of swaging tests to determine workability are listed in table VI. Swaging attempts were made only at elevated temperatures. A 2200° F temperature was set in the majority of cases. This temperature was chosen since microstructural studies indicated that it was close to, but still below, the temperature where melting of minor constituents in the alloy might occur. Experimental difficulties accounted for setting a 2000° and a 2300° F swaging temperature in two instances. The swaging temperature was probably reduced appreciably during the swaging operation because of the disparity in temperature and mass between the dies and the swage bar. The percent reduction in area was approximately 7.5 percent or greater in all cases. There was a difference in the degree of swageability of the alloys as is evidenced by the good and poor designations cited in the table. A good designation indicates that cracks were either nonexistent or few, and apparent only after zyglo inspection. A poor designation indicates that the cracks were numerous and readily apparent without zyglo inspection. Only two compositions could be classified as good.

The strongest alloy (Mo-4-W + 2.5 V + 0.125 C) was evaluated to determine its workability by rolling, and the results of these tests are summarized in table VII. (This alloy was one of those rated "poor" in swaging). It is apparent from the table that only a few of many possible rolling variables were considered. The data were all obtained with a constant roll speed of 84 feet per minute. It was possible to make only minimal reductions (2 percent) without initiating immediately apparent cracks. Attempts to further reduce the rolling samples by a second pass through the rolls were not successful.

On the basis of the data, the alloys described herein would appear to be essentially usable only as cast alloys, although a limited degree of swageability was demonstrated in some cases.

Hardness Data

Hardness data for the alloys investigated are summarized in table VIII; average values are shown. No significant trend is apparent either with variations in composition from the basic alloy or for the homogenization treatment employed. Rockwell "C" values were converted to the nearest whole number from the experimentally obtained Rockwell "A" values by using a standard conversion table.

Metallographic Studies

Photomicrographs of the strongest vanadium-plus-carbon modification of the basic NASA alloy (basic alloy + 2.5 V + 0.125 C) and the strongest tungsten-vanadium-carbon modification of the basic NASA alloy (Mo-4-W + 2.5 V + 0.125 C) are presented in figures 6 and 7, respectively, for both the as-cast and homogenized conditions. Two magnifications, X250 and X750, are provided. Both alloys show a fine dispersion of particles throughout the matrix with the tungsten-vanadium-carbon modification providing the finer dispersion.

In order to better understand these alloys, identification of the phases present would appear to be in order for those interested in pursuing their development. This was not done in the present investigation. It may be reasonably postulated that vanadium-bearing carbides are present, since vanadium is a strong carbide former. It is also possible that an intermetallic phase (nickel-aluminum-vanadium) analogous to the gamma prime phase ($Ni_3(Al,Ti)$) formed in nickel-base alloys containing aluminum and titanium is present.

Figures 8 and 9 depict the microstructure in the area of the exposed surfaces of tested stress-rupture bars for the strongest alloy investigated herein (Mo-4-W + 2.5 V + 0.125 C) and the strongest alloy of reference 1 (basic + 1.5 Ti + 0.125 C). Figure 8(a) represents a condition of 1800° F operation; 8(b), 1850° F operation; and 8(c), 1900° F operation for alloy Mo-4-W + 2.5 V + 0.125 C. Figure 9(a) represents a condition of 1800° F operation, and figure 9(b) represents 1850° F operation for the basic + 1.5 Ti + 0.125 C alloy.

The photomicrographs illustrate typical examples of the subscales formed during stress-rupture tests. It should be noted that in all cases the specimens were run to failure in air-atmosphere furnaces, and the test time at a given temperature was markedly different for the two alloys. If it were assumed that the subscales shown are oxides, it is apparent from the figures that there is more oxide penetration in the vanadium modified alloy than in the titanium modified alloy. This is not surprising in view of the relatively poor oxidation resistance of vanadium above 1200° F. There is certainly no evidence of catastrophic oxidation. Whether the presence of the subscale is detrimental (from the standpoint of rupture properties, at least up to 1900° F) cannot be established on the basis of the data obtained herein. The greatly improved stress-rupture properties of the vanadium-bearing alloys as opposed to the titanium-bearing alloys would tend to indicate that any detrimental effect due to susceptibility to oxidation is more than compensated for by other factors.

SUMMARY OF RESULTS

The following results were obtained from the continued study of advanced-temperature nickel-base alloys to investigate vanadium additives:

1. A series of high-strength, high-temperature alloys was evolved consisting of vanadium, carbon, and tungsten modifications of a basic

alloy having a composition of 79 percent nickel, 8 percent molybdenum, 6 percent chromium, 6 percent aluminum, and 1 percent zirconium.

2. The strongest vanadium-carbon modification of the basic alloy (basic + 2.5 V + 0.125 C) displayed an as-cast rupture life of 565 hours at 1800° F and 15,000-psi stress. The strongest tungsten-vanadium-carbon basic alloy modification (Mo-4-W + 2.5 V + 0.125 C) displayed as-cast rupture lives of 768, 301, and 101 hours at 1800°, 1850°, and 1900° F and 15,000-psi stress in single tests.

3. All the alloys demonstrated good impact resistance. Most of them exceeded 62.5 inch-pounds impact resistance.

4. Both the stress-rupture and impact properties of the strongest vanadium-carbon basic alloy modification and the strongest vanadium-carbon-tungsten basic alloy modification represent substantial improvements over the strongest alloy (basic + 1.5 Ti + 0.125 C) developed in this series in an earlier investigation.

5. The alloys evolved in this investigation must be considered as being essentially cast alloys, although a limited degree of workability was demonstrated in a few instances by swaging tests.

6. There was no evidence of catastrophic oxidation in the vanadium-bearing alloys after prolonged stress-rupture testing in air at 1800°, 1850°, and 1900° F.

7. None of the alloys required vacuum-melting techniques. All were melted under an inert gas blanket and were poured in air.

Lewis Research Center
National Aeronautics and Space Administration
Cleveland, Ohio, October 23, 1959

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TABLE I. - NOMINAL COMPOSITIONS OF ALLOYS INVESTIGATED

Alloy	Composition, weight percent							
	Ni	Mo	Cr	Al	Zr	W	V	C
Basic alloy with vanadium and carbon modifications								
Basic + 1.0 V + 0.125 C	77.875	8	6	6	1	-	1.0	0.125
Basic + 1.5 V + 0.125 C	77.375					-	1.5	.125
Basic + 2.5 V + 0.125 C	76.375					-	2.5	.125
Basic + 3.5 V + 0.125 C	75.375					-	3.5	.125
Basic + 5.0 V + 0.125 C	73.875					-	5.0	.125
Basic + 1.0 V + 0.25 C	77.75					-	1.0	.25
Basic + 1.5 V + 0.25 C	77.25					-	1.5	.25
Basic + 2.5 V + 0.25 C	76.25					-	2.5	.25
Basic + 3.5 V + 0.25 C	75.25					-	3.5	.25
Basic + 5.0 V + 0.25 C	73.75					-	5.0	.25
Basic alloy with tungsten, vanadium, and carbon modifications								
Mo-4-W	79.00	4	6	6	1	4	---	-----
Mo-2-W + 2.5 V + 0.125 C	76.375	6				2	2.5	0.125
Mo-4-W + 2.5 V + 0.125 C	76.375	4				4	2.5	.125
Mo-6-W + 2.5 V + 0.125 C	76.375	2				6	2.5	.125
Mo-8-W + 2.5 V + 0.125 C	76.375	-				8	2.5	.125

TABLE II. - COMPOSITIONS OF ALLOYS FROM RANDOMLY SELECTED HEATS AS DETERMINED BY CHEMICAL ANALYSIS

Alloy	Composition, weight percent							
	Ni	Mo	Cr	Al	V	C	Zr	W
Basic + 1.0 V + 0.125 C	Bal.[a]	8.08	5.61	5.44	0.90	0.17	0.87	----
Basic + 1.5 V + 0.125 C		8.18	5.67	5.24	1.30	.14	.89	----
Basic + 2.5 V + 0.125 C		8.25	5.61	5.69	2.20	.13	.53	----
Basic + 3.5 V + 0.125 C		8.23	5.58	5.70	3.22	.14	.64	----
Basic + 1.0 V + 0.25 C		8.23	5.61	5.37	.96	.25	.86	----
Basic + 1.5 V + 0.25 C		8.33	5.64	5.56	1.40	.26	.83	----
Basic + 2.5 V + 0.25 C		8.21	5.59	5.41	2.19	.26	.67	----
Basic + 3.5 V + 0.25 C		8.39	5.51	5.60	3.10	.26	.57	----
Basic + 5.0 V + 0.25 C		8.11	5.55	5.55	4.34	.27	1.00	----
Mo-4-W		4.06	5.49	5.36	----	----	.92	3.56
Mo-4-W + 2.5 V + 0.125 C		4.07	5.54	5.57	2.28	.16	.63	3.45

[a]Balance obtained by subtraction.

TABLE III. - SUMMARY OF STRESS-RUPTURE LIVES AT 15,000-PSI STRESS

Alloy	Condition	Temperature, °F	Life, hr
Basic	As cast	1800	18.85
	Homogenized	1800	45.7
Basic + 0.125 C	As cast	1800	73.3
	Homogenized	1800	132.7
Basic + 0.25 C	As cast	1800	113.3
	Homogenized	1800	131.0
Basic + 1.0 V + 0.125 C	As cast	1800	307.2
	Homogenized	1800	172.3
Basic + 1.5 V + 0.125 C	As cast	1800	438.7
	Homogenized	1800	260.2
Basic + 2.5 V + 0.125 C	As cast	1800	564.65
	Homogenized	1800	412.1
	As cast	1850	82.2
	As cast	1900	25.3
Basic + 3.5 V + 0.125 C	Homogenized	1800	428.8
Basic + 5.0 V + 0.125 C	As cast	1800	461.15
	Homogenized	1800	438.3
Basic + 1.0 V + 0.25 C	As cast	1800	180.0
	Homogenized	1800	234.55
Basic + 1.5 V + 0.25 C	As cast	1800	187.8
	As cast	1800	205.35
	Homogenized	1800	264.6
Basic + 2.5 V + 0.25 C	As cast	1800	347.8
	Homogenized	1800	150.75
	Homogenized	1800	184.35
Basic + 3.5 V + 0.25 C	As cast	1800	404.8
	Homogenized	1800	281.35
Basic + 5.0 V + 0.25 C	As cast	1800	431.6
	Homogenized	1800	320.45
	As cast	1900	25.9
Mo-4-W	As cast	1800	26.4
	Homogenized	1800	14.45
Mo-4-W + 2.5 V + 0.125 C	As cast	1800	767.85
	Homogenized	1800	745.2
	As cast	1850	300.8
	As cast	1900	101.1
Mo-2-W + 2.5 V + 0.125 C	As cast	1900	31.1
Mo-6-W + 2.5 V + 0.125 C	As cast	1900	44.8
Mo-8-W + 2.5 V + 0.125 C	As cast	1900	21.2

TABLE IV. - TENSILE PROPERTIES OF NASA ALLOYS COMPARED WITH

COMMERCIAL HIGH-TEMPERATURE NICKEL-BASE ALLOYS

Alloy	Condition	Ultimate tensile strength, psi		Elongation, percent		Reduction of area, percent	
		Room temp.	1800° F	Room temp.	1800° F	Room temp.	1800° F
Basic + 2.5 V + 0.125 C	As cast	104,000	60,900	11.7	6.8	27.0	12.1
Mo-4-W + 2.5 V + 0.125 C	As cast	113,000	66,900	1.6	4.1	0.6	4.7
Nicrotung[a]	As cast	130,000	67,000	5.0	6.0	----	----
Udimet 700 (forged)[b]	Heat-treated	202,000	52,000	16.0	27.0	20.0	27.0

[a]Data from Westinghouse preliminary data folder.

[b]Data from Kelsey-Hayes Company preliminary data folder.

E-532

CX-3 back

TABLE V. - IMPACT RESISTANCE OF NASA ALLOYS COMPARED WITH COMMERCIAL HIGH-TEMPERATURE ALLOYS

Alloy	Room temperature impact resistance, in.-lb	
	As cast	Homogenized
Basic	51.4, 23.0, >62.5, >62.5	>62.5, >62.5, >62.5
Basic + 1.0 V + 0.125 C	>62.5, >62.5	>62.5, >62.5
Basic + 1.5 V + 0.125 C		>62.5, >62.5
Basic + 2.5 V + 0.125 C		>62.5, 32.4
Basic + 3.5 V + 0.125 C		>62.5, >62.5
Basic + 1.0 V + 0.25 C		50.0, 62.0
Basic + 1.5 V + 0.25 C		>62.5, 47.0
Basic + 2.5 V + 0.25 C	>62.5, 45.3	61.5, 49.0
Basic + 3.5 V + 0.25 C	46.6, 62.0	>62.5, 16.5
Basic + 5.0 V + 0.25 C	23.3, 17.2	-----------
Mo-4-W	>62.5, 49.0	>62.5, >62.5
Mo-4-W + 2.5 V + 0.125 C	>62.5, >62.5	>62.5, 49.0
Nicrotung	>62.5, >62.5	----------
X-40	48.1, >62.5, 55.0	----------

TABLE VI. - SUMMARY OF SWAGING DATA FOR NASA ALLOYS IN AS-CAST CONDITION

Alloy	Swage temperature, °F	Reduction of area, percent	Swageability
Basic + 1.0 V + 0.125 C	2300	7.5	Poor
Basic + 1.5 V + 0.125 C	2000	7.5	
Basic + 2.5 V + 0.125 C	2200	7.9	
Basic + 3.5 V + 0.125 C		7.7	
Basic + 5.0 V + 0.125 C		8.8	
Basic + 1.0 V + 0.25 C		7.5	Good
Basic + 1.5 V + 0.25 C		7.7	Good
Basic + 2.5 V + 0.25 C		7.5	Poor
Basic + 3.5 V + 0.25 C		7.7	
Basic + 5.0 V + 0.25 C		17.7	
Mo-4-W		7.7	
Mo-4-W + 2.5 V + 0.125 C		13.0	

TABLE VII. - SUMMARY OF ROLLING DATA FOR

AS-CAST ALLOY Mo-4-W + 2.5 V + 0.125 C

Specimen	Temperature, °F	Reduction, percent	Pass	Results
1	Room	3	1	Poor
2	Room	6		
3	1900	5		
4	1950	4.5		
5	2000	4		
6	2050	2		Good
	2050	16.5	2	Poor
7	2050	1.5	1	Good
	2050	10.5	2	Poor

TABLE VIII. - SUMMARY OF HARDNESS DATA FOR NASA ALLOYS

Alloy	Condition	Average hardness, Rockwell-	
		A (a)	C (b)
Basic	As cast Homogenized	65.9 67.8	31 35
Basic + 1.0 V + 0.125 C	As cast Homogenized	68.7 68.2	36 35
Basic + 1.5 V + 0.125 C	As cast Homogenized	68.8 69.8	37 39
Basic + 2.5 V + 0.125 C	As cast Homogenized	69.8 69.5	39 38
Basic + 3.5 V + 0.125 C	As cast Homogenized	69.9 70.7	39 41
Basic + 1.0 V + 0.25 C	As cast Homogenized	68.4 68.3	36 36
Basic + 1.5 V + 0.25 C	As cast Homogenized	68.7 68.9	36 37
Basic + 2.5 V + 0.25 C	As cast Homogenized	69.1 69.4	37 38
Basic + 3.5 V + 0.25 C	As cast Homogenized	68.0 68.9	35 37
Basic + 5.0 V + 0.25 C	As cast	69.0	37
Mo-4-W	As cast Homogenized	68.0 68.0	35 35
Mo-4-W + 2.5 V + 0.125 C	As cast Homogenized	67.9 69.5	35 38
Mo-2-W + 2.5 V + 0.125 C	As cast	68.1	35
Mo-6-W + 2.5 V + 0.125 C	As cast	67.6	34
Mo-8-W + 2.5 V + 0.125 C	As cast	68.0	35

[a]Rockwell "A" results are average of at least three tests.

[b]Converted from Rockwell "A" values.

E-532

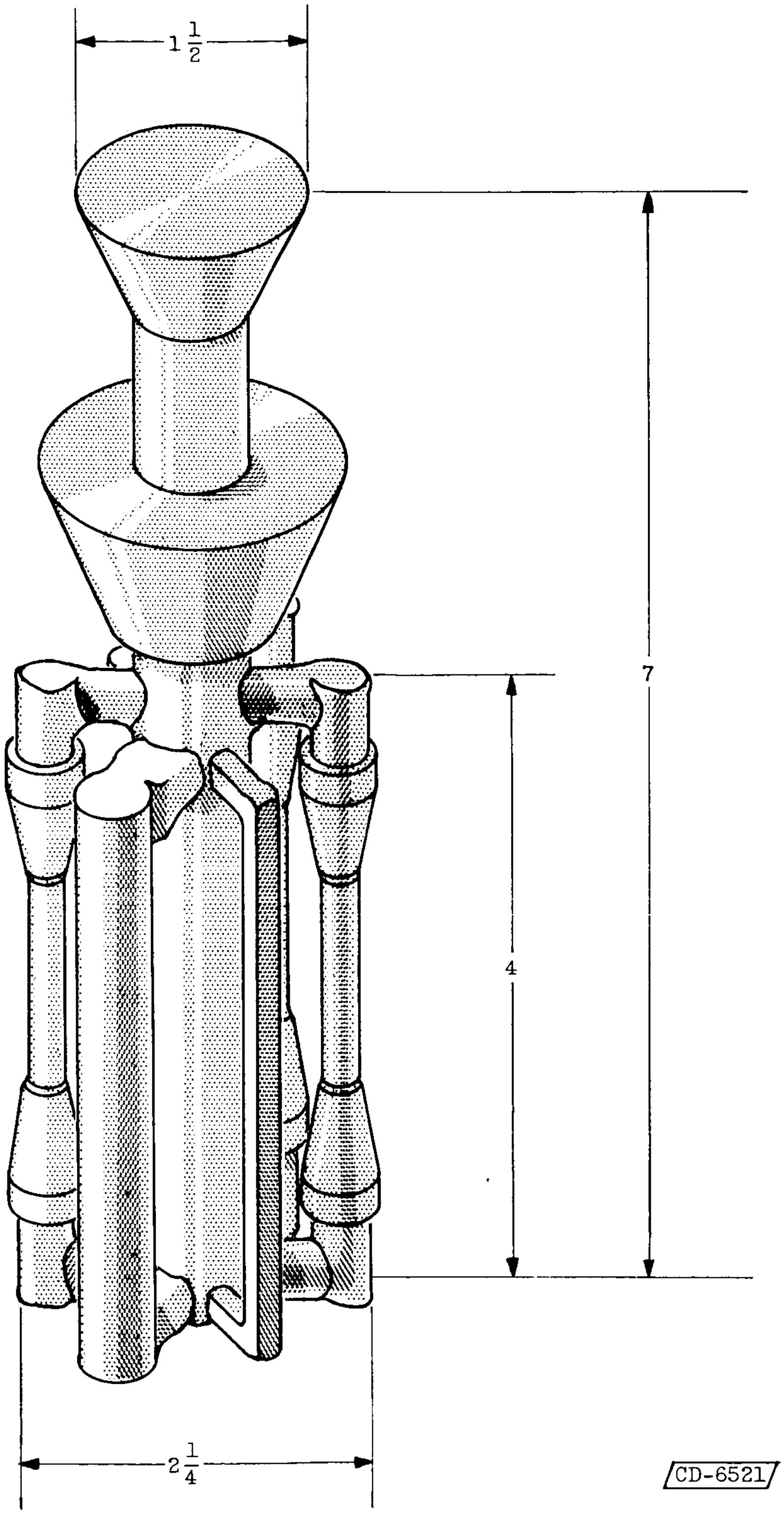


Figure 1. - Assembly of wax patterns for stress-rupture, swage, and impact bars. (All dimensions in inches.)

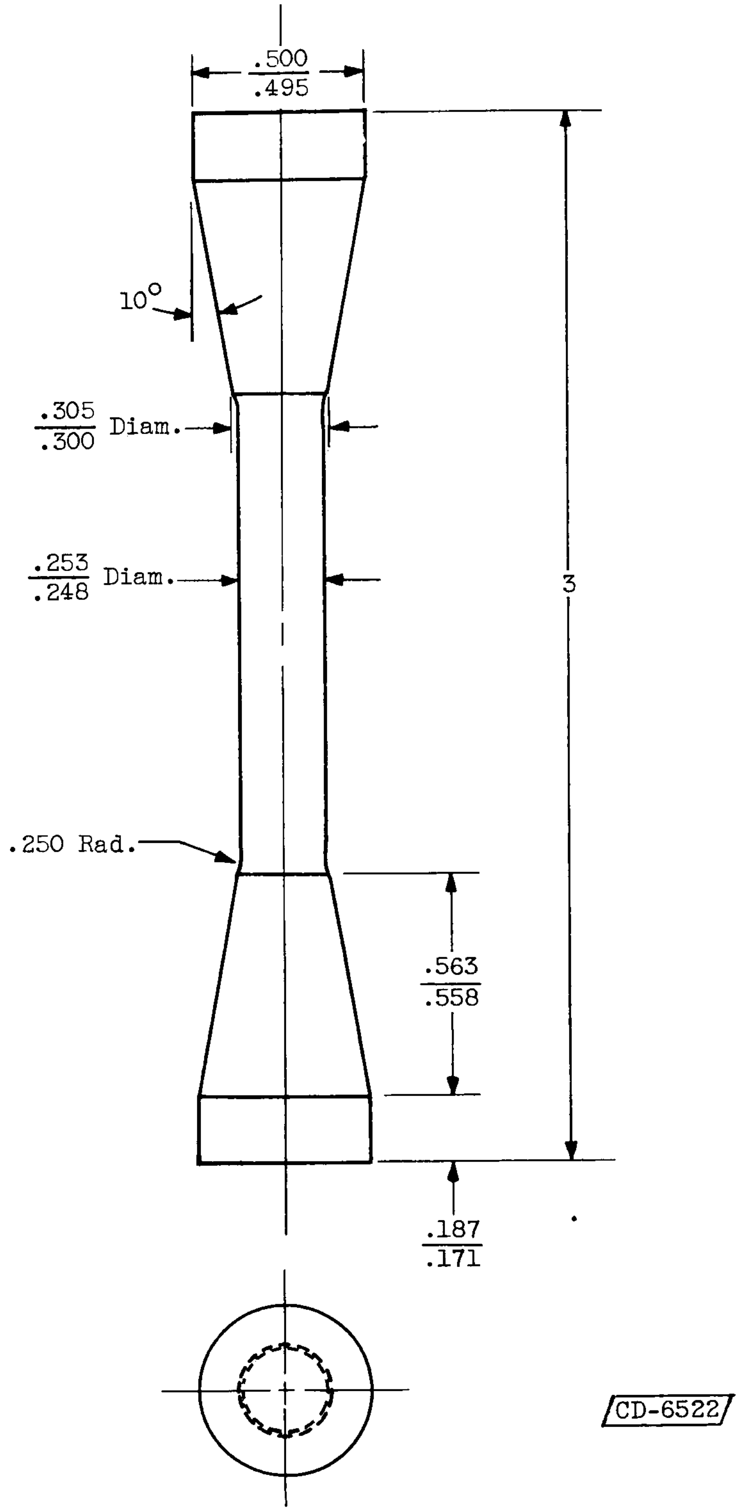


Figure 2. - Dimensions of tensile and stress-rupture bars. (All dimensions in inches.)

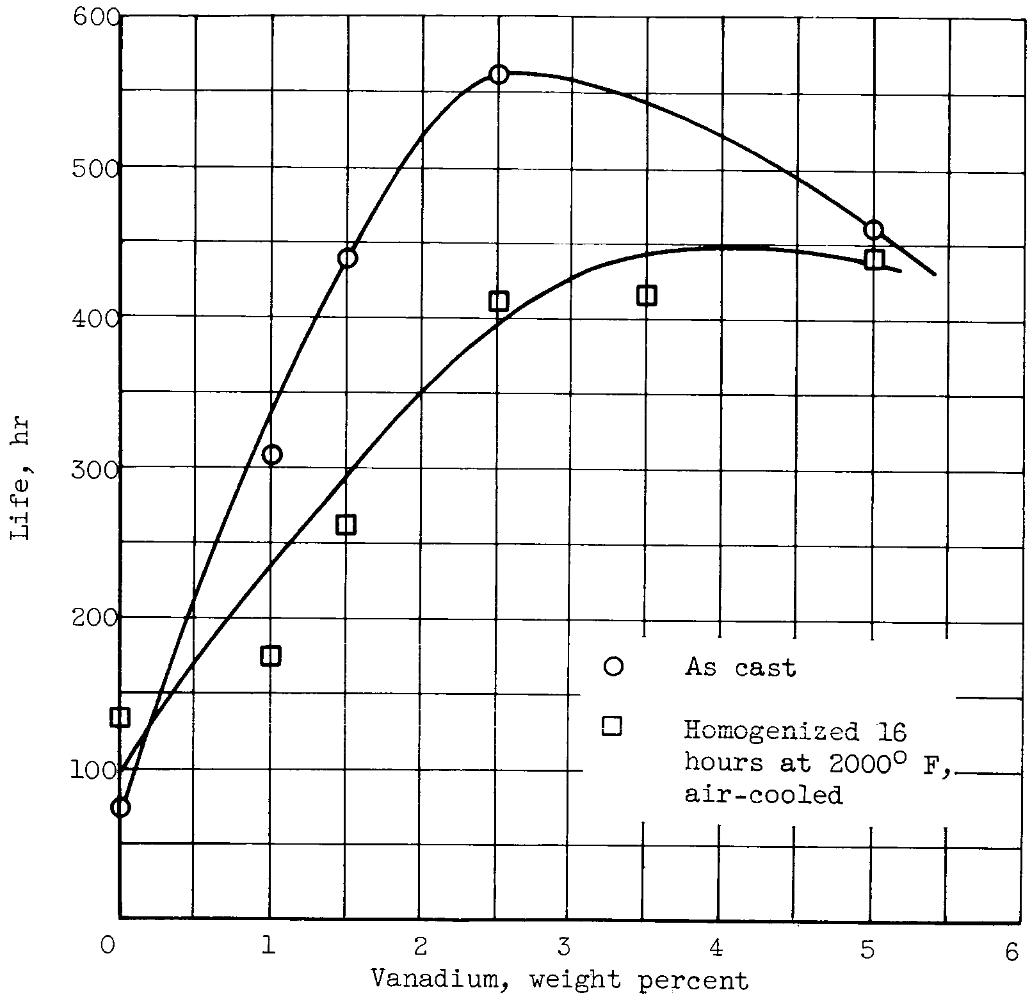


Figure 3. - Effect of vanadium plus 0.125-percent-carbon additions on 1800° F life of basic NASA alloy at 15,000-psi stress.

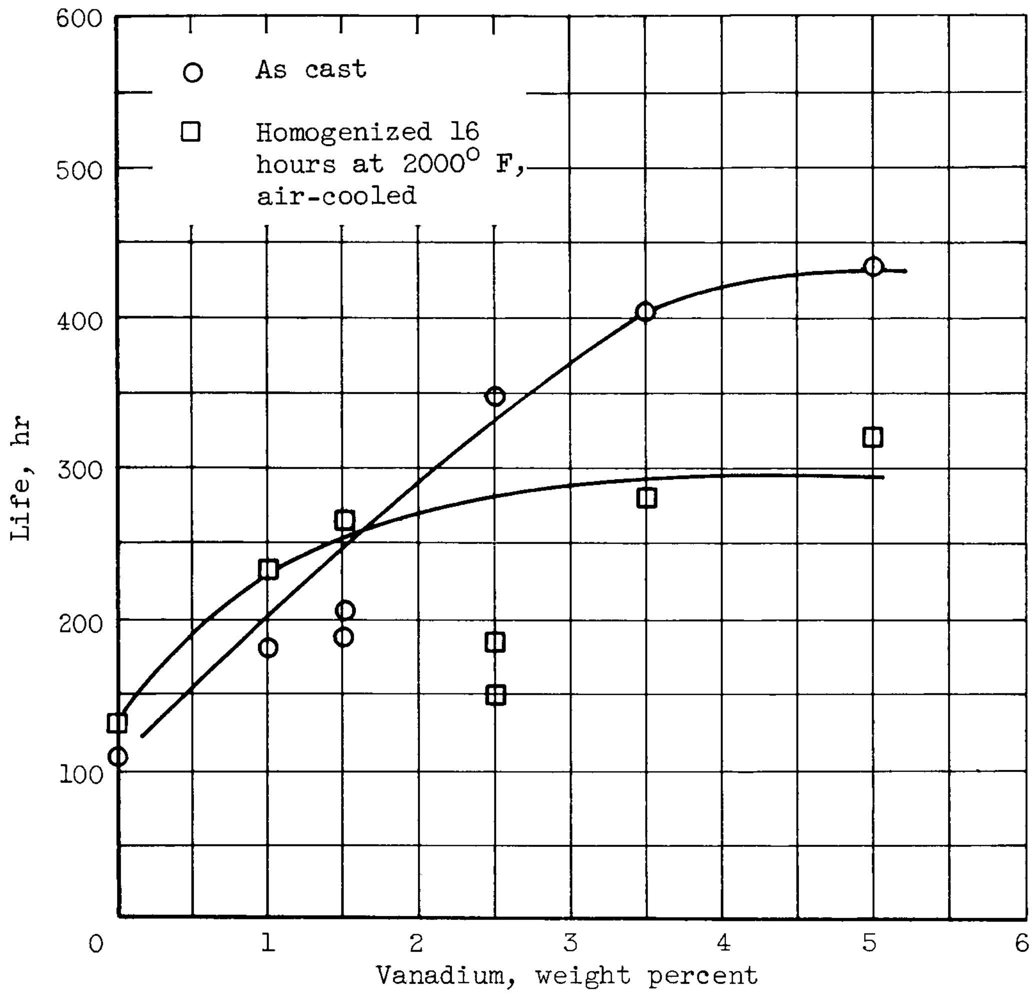


Figure 4. - Effect of vanadium plus 0.25-percent-carbon additions on 1800° F life of basic NASA alloy at 15,000-psi stress.

E-532

CX-4 back

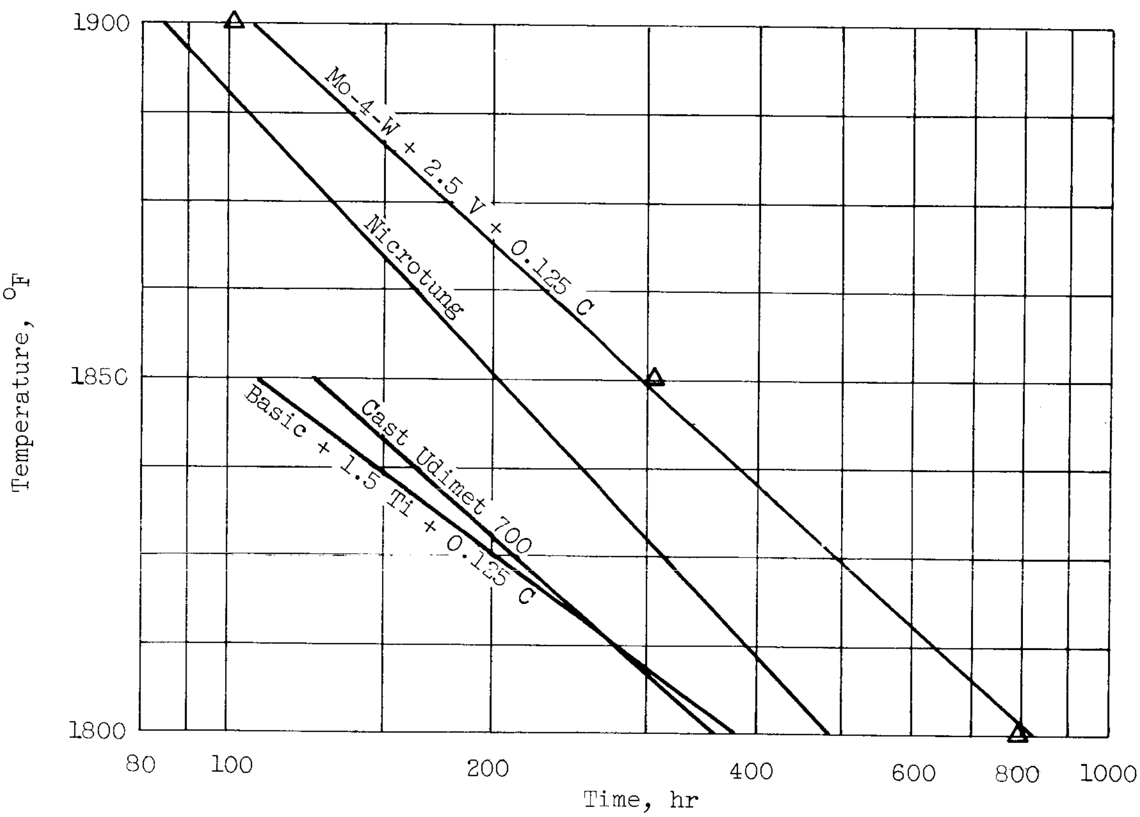
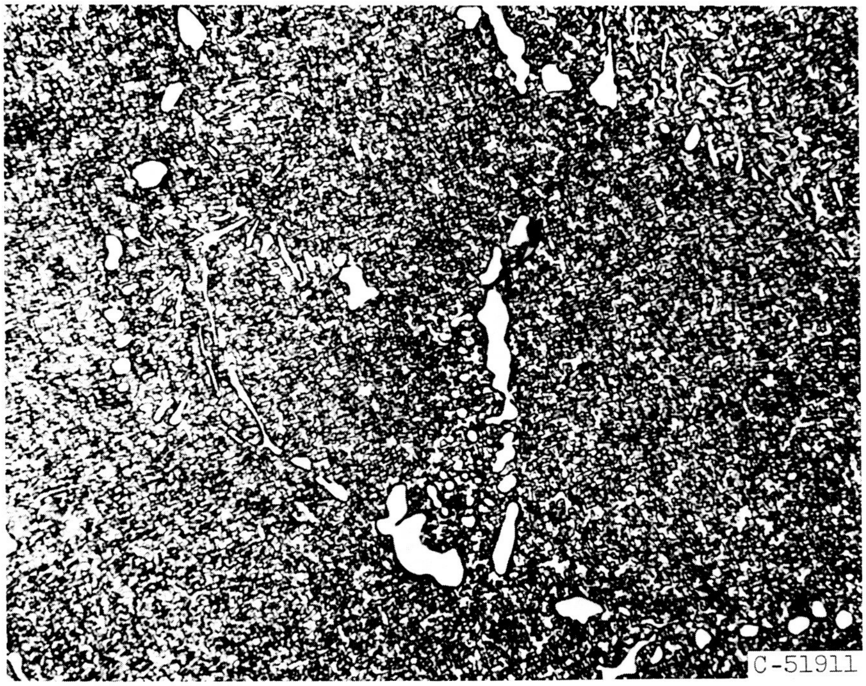


Figure 5. - Stress-rupture comparison at 15,000-psi stress of recent commercial alloys and strongest NASA alloys.

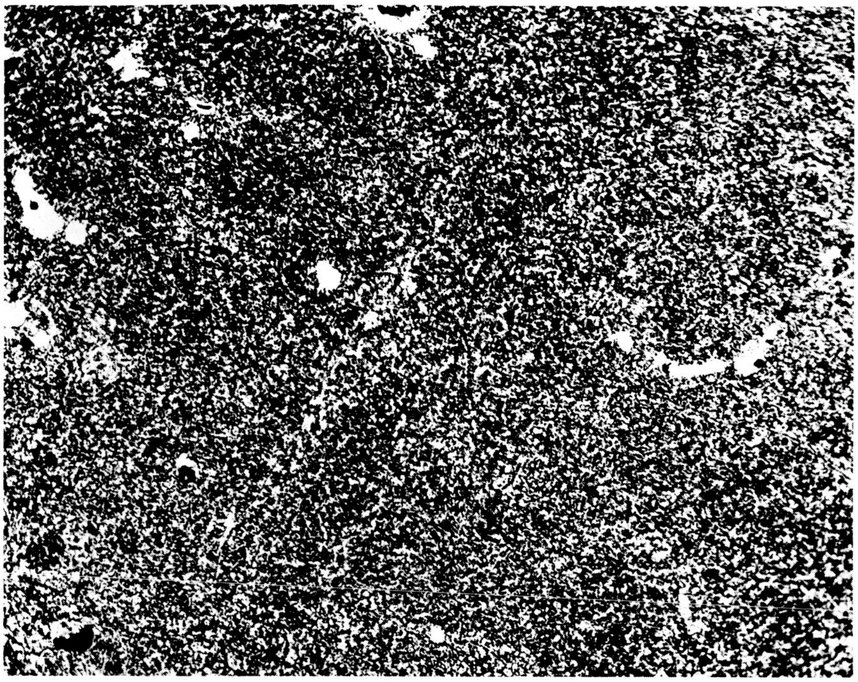
X250



X750

(a) Basic alloy + 2.5 V + 0.125 C, as-cast.

Figure 6. - Microstructures of vanadium-carbon modification of basic alloy.



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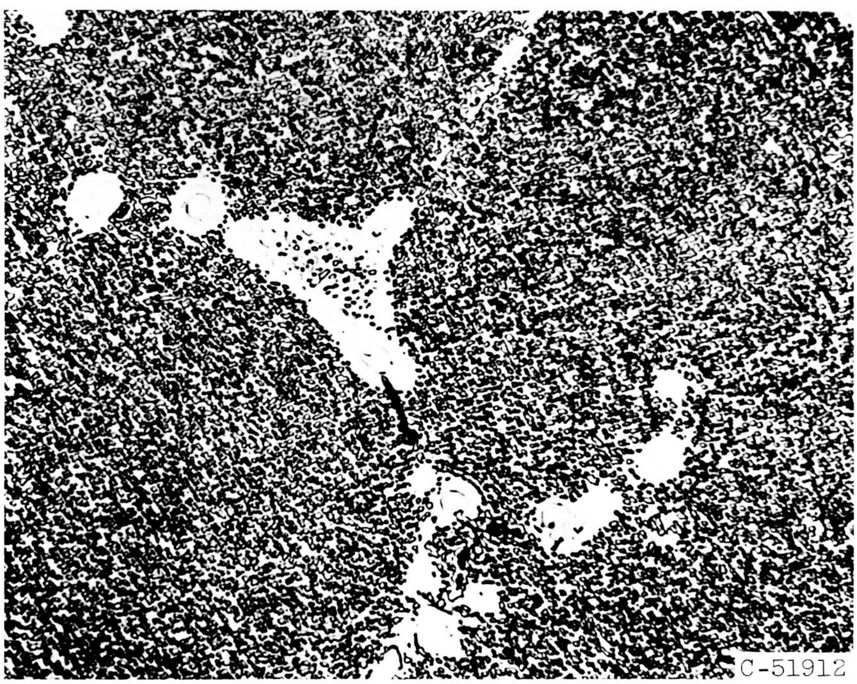
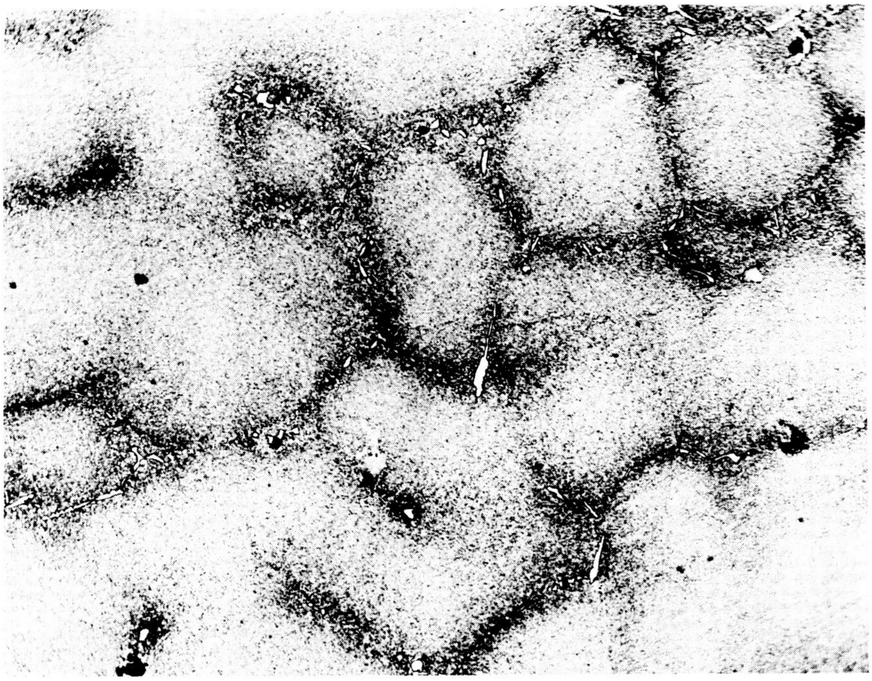


X750

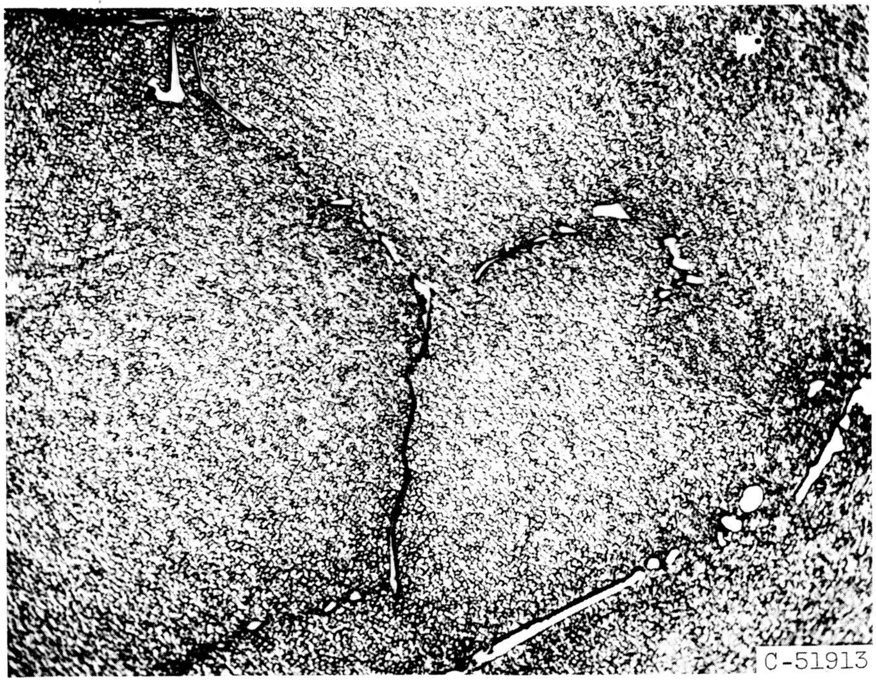
(b) Basic + 2.5 V + 0.125 C, heat-treated 16 hours at 2000° F.

Figure 6. - Concluded. Microstructures of vanadium-carbon modification of basic alloy.

E-532



X250



X750

(a) Alloy Mo-4-W + 2.5 V + 0.125 C, as-cast.

Figure 7. Microstructures of tungsten-vanadium-carbon modification of basic alloy.

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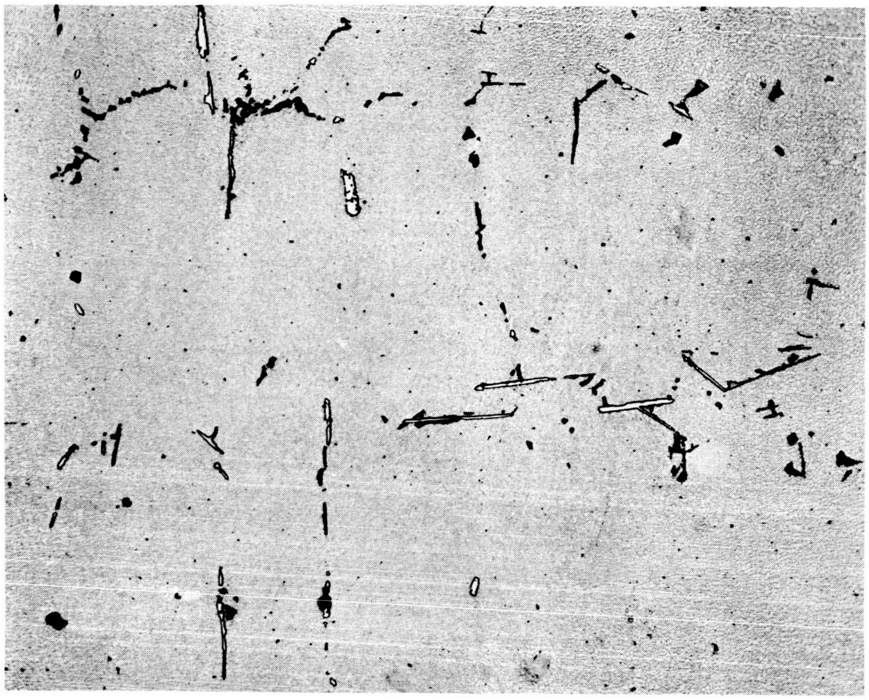
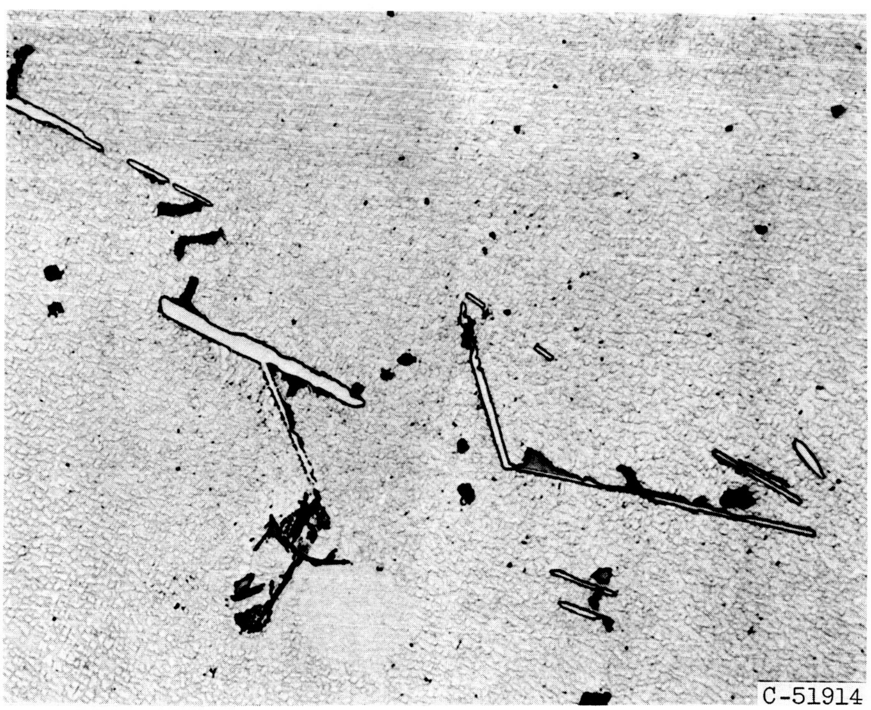


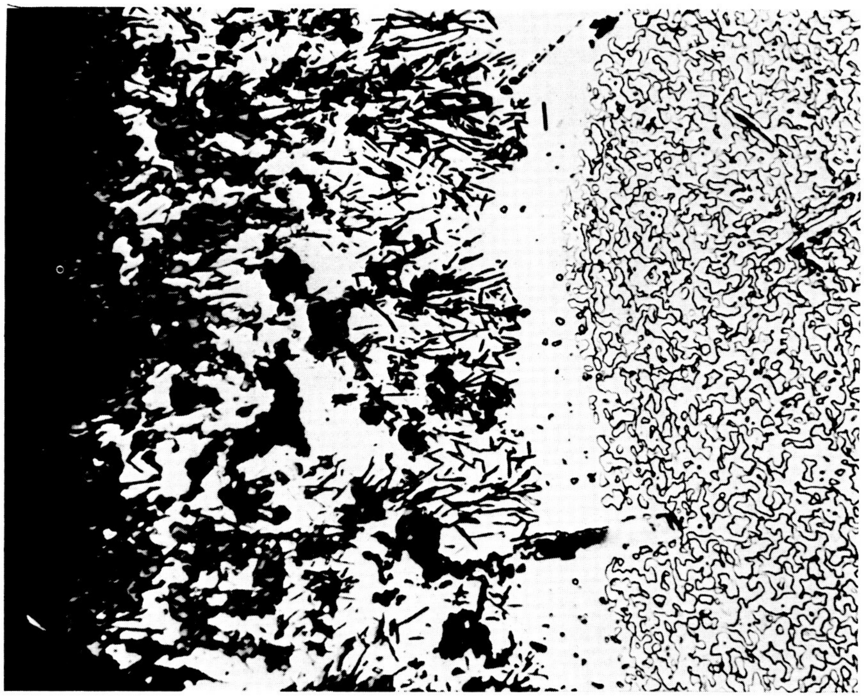
X250



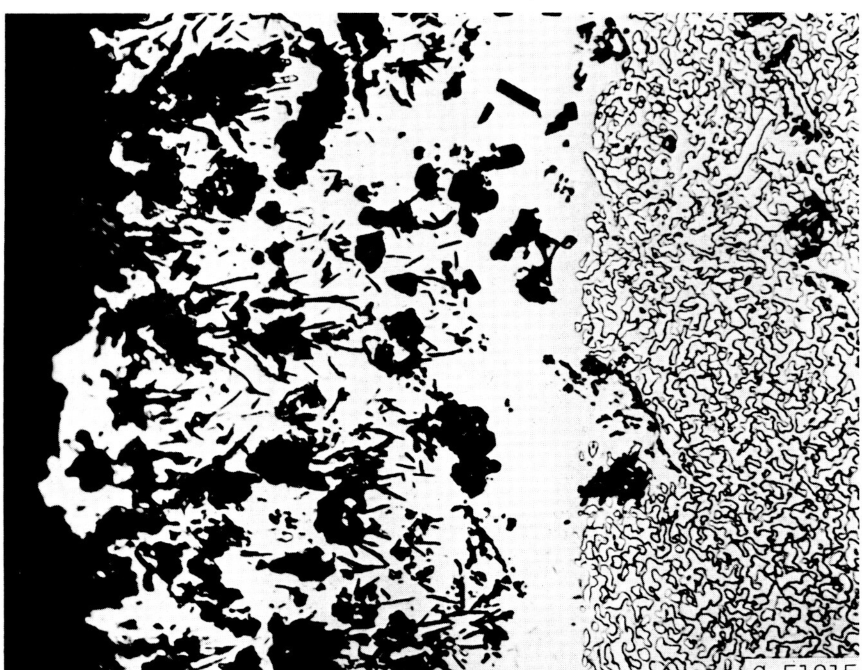
X750

(b) Alloy Mo-4-W + 2.5 V + 0.125 C, heat-treated 16 hours at 2000° F.

Figure 7. - Concluded. Microstructures of tungsten-vanadium-carbon modification of basic alloy.



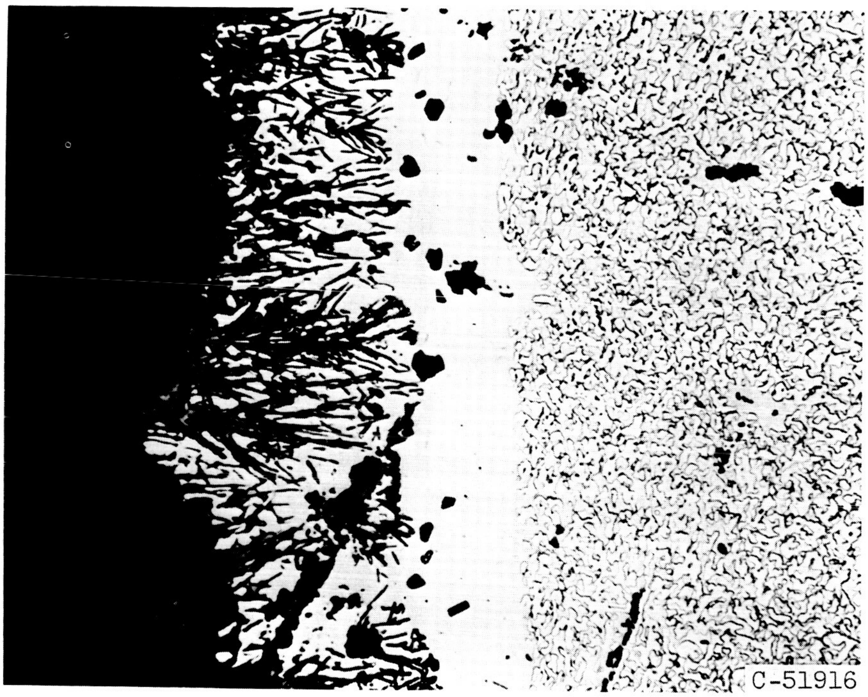
(a) 767.85 Hours at 1800° F.



(b) 300.8 Hours at 1850° F.

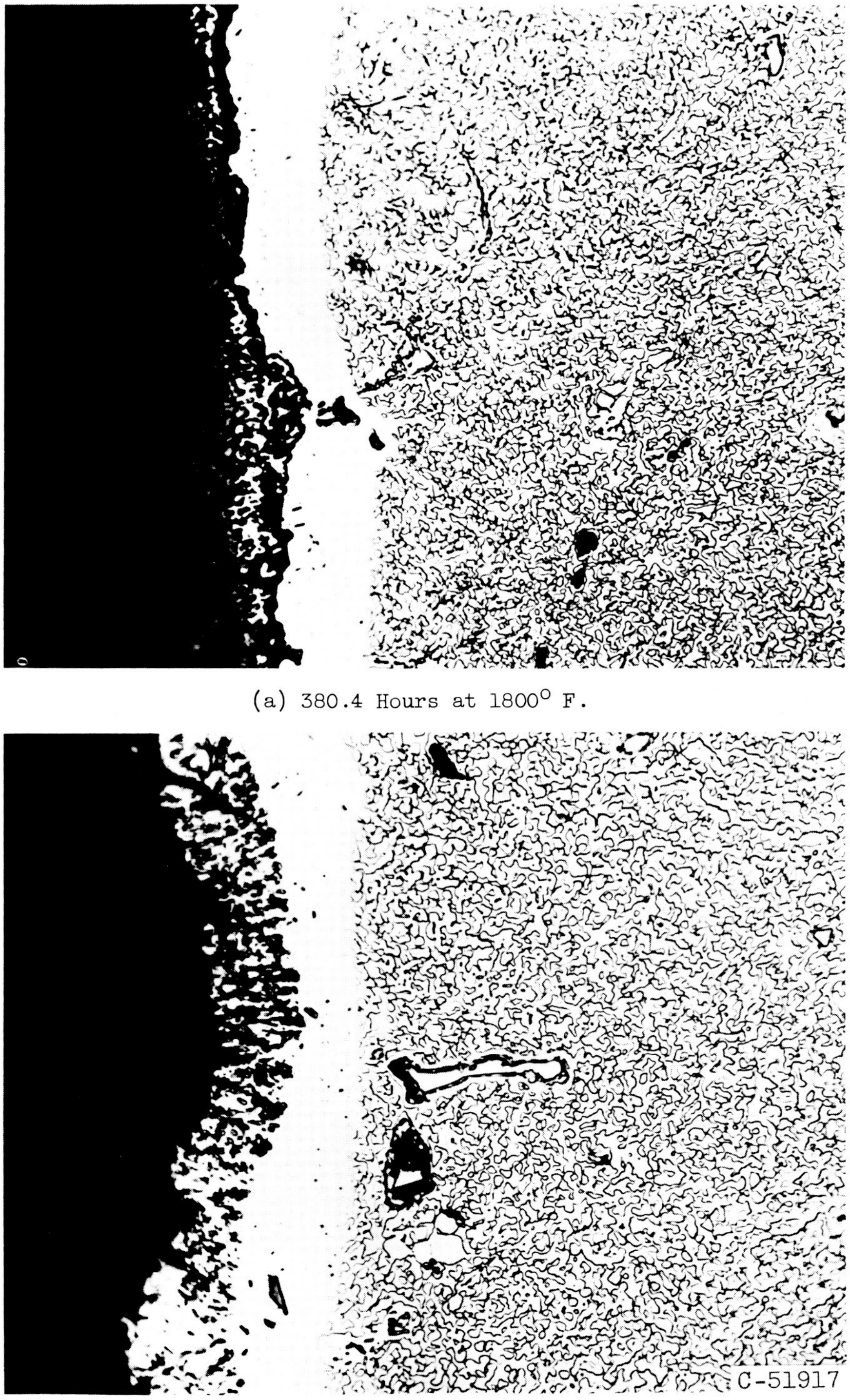
Figure 8. - Microstructures of tested stress-rupture bars of tungsten-vanadium-carbon modification of basic alloy (Mo-4-W + 2.5 V + 0.125 C) in vicinity of exposed surfaces. X750.

E-532



(c) 101.1 Hours at 1900° F.

Figure 8. - Concluded. Microstructures of tested stress-rupture bars of tungsten-vanadium-carbon modification of basic alloy (Mo-4-W + 2.5 V + 0.125 C) in vicinity of exposed surfaces. X750.



(a) 380.4 Hours at 1800° F.

(b) 106.65 Hours at 1850° F.

Figure 9. - Microstructures of tested stress-rupture bars of titanium-carbon modification of basic alloy (basic + 1.5 Ti + 0.125 C) in vicinity of exposed surfaces. X750.